

## DRAFT

### Standard Format and Guidance for AOAC Standard Method Performance Requirement (SMPR) Documents (Version 9; September 7, 2010)

#### AOAC SMPR 2010.XXX

**Method Name:** must include the analyte(s), matrix(-es), and analytical technique (unless the SMPR is truly intended to be independent of the analytical technology). The method name may refer to a "common" name (e.g. "Kjeldahl" method).

**Approved by:** Stakeholder Panel or Expert Review Panel name

**Final version date:** date

**Effective date:** date

- 1. Intended Use:** Additional information about the method and conditions for use.
- 2. Applicability:** List matrixes if more than one. Provide details on matrix such as specific species for biological analytes, or IUPAC<sup>1</sup> nomenclature and CAS<sup>2</sup> registry number for chemical analytes. Specify the form of the matrix such as raw, cooked, tablets, powders, etc.
- 3. Analytical Technique:** Provide a detailed description of the analytical technique if the SMPR is to apply to a specific analytical technique; or state that the SMPR applies to any method that meets the method performance requirements.
- 4. Definitions:** List and define terms used in the performance parameter table (see table 2 for list of standard terms)
- 5. Method Performance Requirements:** List the performance parameters and acceptance criteria appropriate for each method/analyte/matrix. See table 1 for appropriate performance requirements.  
  
If more than one analyte/matrix, and if acceptance criteria differ for analyte/matrix combinations then organize a table listing each analyte/matrix combination and its minimum acceptance criteria for each performance criteria.
- 6. System suitability tests and/or analytical quality control:** Describe minimum system controls and QC procedures.
- 7. Reference Method(s):** Identify the appropriate reference method if one exists, or state that a reference method does not exist.
- 8. Reference Material(s):** Identify the appropriate reference materials if they exist, or state that reference materials are not available.
- 9. Validation Guidance:** Qualify the method into one of the method classifications in Table 1. which provides general recommendations regarding validation of the method. Validation study protocols should be provided as an annex to the SMPR. Identify which studies should be carried out at the method developers site (single laboratory validation); and the independent laboratory (for the *Performance Tested Methods* program).
- 10. Maximum Time-To-Determination:** Maximum allowable time to complete an analysis starting from the test portion preparation to final determination or measurement.

<sup>1</sup> International Union of Pure and Applied Chemistry (IUPAC)

<sup>2</sup> Chemical Abstracts Service

## Annex I: Validation Study Protocols

(Required for all SMPRs)

**Introduction:** Provide basic information about the type of methods that are applicable to the validation outline; and general information about the levels of validation required for the SMPR method.

**Level 1: Method Developer Validation Study Protocol.** Describe the studies that a method developer must carry out to demonstrate that a candidate method meets the performance parameters specified in an SMPR. Refer to Table 3 for the recommended studies.

**Level 2: Independent Laboratory Validation Study Protocol.** Describe the studies that an independent laboratory must carry out to demonstrate that a candidate method meets the performance parameters specified in an SMPR. Refer to Table 3 for the recommended studies

**Level 3: Collaborative Study Protocol.** Describe the collaborative study that a Method Developer / Study Director must carry out to demonstrate that a candidate method meets the performance parameters specified in an SMPR.

### ALL VALIDATION PROTOCOLS MUST INCLUDE THE FOLLOWING STATEMENTS:

Method developers seeking to submit data to the *Official Methods* or *Performance Tested Methods* programs must prepare, submit, and obtain approval of their individualized validation study protocol.

An approved protocol is valid for one year from its approval date. Validation protocols with approval dates more than one year must be submitted for re-approval.

Table 1: Performance Requirements

Classifications of Methods <sup>9</sup>						
	Quantitative Method (main component <sup>1</sup> )	Quantitative Method (trace or contaminant <sup>2</sup> )	Qualitative Method (main component <sup>1</sup> )	Qualitative Method (trace or contaminant <sup>2</sup> )	Identification Method	
Parameters	Single laboratory validation	Reference Method Comparison <sup>3</sup> Applicable Range Bias <sup>4</sup> Precision Recovery	Reference Method Comparison <sup>3</sup> Applicable Range Bias <sup>4</sup> Precision Recovery Limit of Detection (LOD) Limit of Quantitation (LOQ)	Reference Method Comparison <sup>3</sup> Inclusivity/Selectivity Exclusivity/Specificity Environmental Interference Laboratory Variance Bias <sup>4</sup> Probability of Detection <sup>6</sup>	Reference Method Comparison <sup>3</sup> Inclusivity/Selectivity Exclusivity/Specificity Environmental Interference Laboratory Variance Bias <sup>4</sup> Probability of Detection (POD) at the AMDL <sup>8</sup>	Reference Method Comparison <sup>3</sup> Inclusivity/Selectivity Exclusivity/Specificity Precision Environmental Interference Bias <sup>4</sup>
	Independent	TBD <sup>5</sup>	TBD <sup>5</sup>	TBD <sup>5</sup>	Probability of Detection (POD) at the AMDL <sup>8</sup>	Bias <sup>4</sup>
	Collaborative Study	Reproducibility	Reproducibility	POD (0) POD (c) Laboratory Probability of Detection <sup>8</sup>	POD (0) POD (c) Laboratory Probability of Detection <sup>7</sup>	

Notes:

1. ≥100 g/kg
2. <100 g/kg
3. If a reference method is available
4. If a reference material is available
5. To be determined by the Topic Advisor/ General Referee
6. At a critical level.
7. If a reference method is available. LPOD = CPOD.
8. Acceptable Minimum Detection Level (AMDL)
9. See Appendix B for additional information on classification of methods.

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**Table 3: Recommendations for Evaluation**

Accuracy	The closeness of agreement between a test result and the accepted reference value. The term accuracy, when applied to a set of test results, involves a combination of random components and a common systematic error or bias component.
Bias (if a reference material is available)	The difference between the expectation of the test results and an accepted reference value. Bias is the total systematic error as contrasted to random error. There may be one or more systematic error components contributing to the bias.
Environmental Interference	Analyze test portions containing a specified concentration of one environmental materials panel member. Materials may be pooled. Consult with AOAC statistician.
Exclusivity	Analyze one test portion containing a specified concentration of one exclusivity panel member. More replicates can be used. Consult with AOAC statistician.
Inclusivity	Analyze one test portion containing a specified concentration of one inclusivity panel member. More replicates can be used. Consult with AOAC statistician.
Limit of Detection (LOD)	Measure 10 blank samples. Calculate the mean average and standard deviation of the results $LOD^4 = \text{average (blank)} + z s_0 \text{ (blank)}$ ; where $s_0 = \text{standard deviation}$ $z = 2 \times \text{Gaussian critical value} = 2 \times 1.645 = 3.3$
Limit of Quantitation (LOQ)	Estimate the LOQ = average (blank) + 10 * s0 (blank); Measure blank samples with analyte at the estimated LOQ. Calculate the mean average and standard deviation of the results Guidance <sup>5</sup> : For ML ≥ 100 ppm (0.1 mg/kg): LOD = ML * 1/5 For ML < 100 ppm (0.1 mg/kg): LOD = ML * 2/5
POD(0)	Use data from collaborative study
POD (c)	

<sup>4</sup> ISO 16140:2003

<sup>5</sup> Codex Alimentarius Codex Procedure Manual

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Precision (repeatability)	Prepare and homogenize 3 unknown samples at different concentrations to represent the full, claimed range of the method. Analyze each unknown sample by the candidate method 7 times, beginning each analysis from weighing out the test portion through to final result with no additional replication (unless stated to do so in the method). All of the analyses for one unknown sample should be performed within as short period of time as is allowed by the method. The second and third unknowns may be analyzed in another short time period. Repeat for each matrix.	
Probability of Detection (POD)	Determine the desired Probability of Detection at a critical concentration. Consult with table 7 to determine the number of test portions required to demonstrate the desired Probability of Detection.	
Recovery	Determined from spiked blanks or samples with at least 7 independent analyses per concentration level at a minimum of 3 concentration levels covering the analytical range. Independent means at least at different times. If no confirmed (natural) blank is available, the average inherent (naturally containing) level of the analyte should be determined on at least 7 independent replicates.	
Relative standard deviation (RSD)	$RSD = s_i \times 100 / \bar{x}$	
Reproducibility (Collaborative study)	Quantitative methods	Recruit 10-12 collaborators Must have 8 valid data sets 2 blind duplicate replicates at five concentrations for each analyte/matrix combination to each collaborator
	Qualitative methods	Recruit 12-15 collaborators Must have 10 valid data sets 6 replicates at five concentrations for each analyte/matrix combination to each collaborator
Standard deviation ( $s_i$ )	$s_i = [\sum(x_i - \bar{x}_i)^2 / n]^{0.5}$	

